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DETERMINATION OF THE WELDABILITY AND ELEVATED TEMPERATURE STABILITY OF REFRACTORY METAL ALLOYS

Second Quarterly Report

by

G. G. Lessmann and D. R. Stoner

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
UNDER CONTRACT NAS3-2540

Astronuclear Laboratory
Westinghouse Electric Corporation

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Astronuclear

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SECOND QUARTERLY REPORT

Covering the Period

September 21, 1963 to December 21, 1963

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2540

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FOREWORD

This report describes work accomplished under Contract NAS 3-2540 during the period September 21, 1963 to December 21, 1963. This program is being administered by R. T. Begley of the Astronuclear Laboratory, Westinghouse Electric Corporation. G. G. Lessmann and D. R. Stoner performed the experimental investigations.

P. E. Moorhead of the Nuclear Power Technology Branch, National Aeronautics and Space Administration, is Technical Manager of this program.

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I. INTRODUCTION

This is the Second Quarterly Progress Report describing work accomplished under Contract NAS 3-2540. The objective of this program is to determine the weldability and long-time elevated temperature stability of promising refractory metal alloys in order to determine those most suitable for use in advanced alkali-metal space electric power systems. A detailed discussion of the program and program objectives was presented in the First Quarterly Report.[1]

A chronological outline of this study is shown in Figure 1, while those alloys to be included in the investigation are listed in Table I. Each alloy will receive essentially the same evaluation, though as the program evolves certain alloys may require tests tailored to unique responses to test conditions. Alloys will be tested in the form of 0.035 inch sheet and 0.375 inch plate, with the exception of W-25% Re and pure W which will be evaluated only in sheet form. The inert gas shielded arc and electron beam welding processes will be used.

Process and test controls employed throughout this program emphasize the important influence of the interstitial elements, carbon, oxygen, and nitrogen, on the properties of refractory metals and their alloys. Stringent test procedures are required, including continuous monitoring of the welding chamber atmosphere for oxygen and water vapor levels, electron beam welding at low pressures in a well-trapped system, aging in furnaces employing hydrocarbon free pumping systems providing pressures less than 10^{-8} Torr, and chemical sampling following successive stages of the evaluation for verification of these process controls.

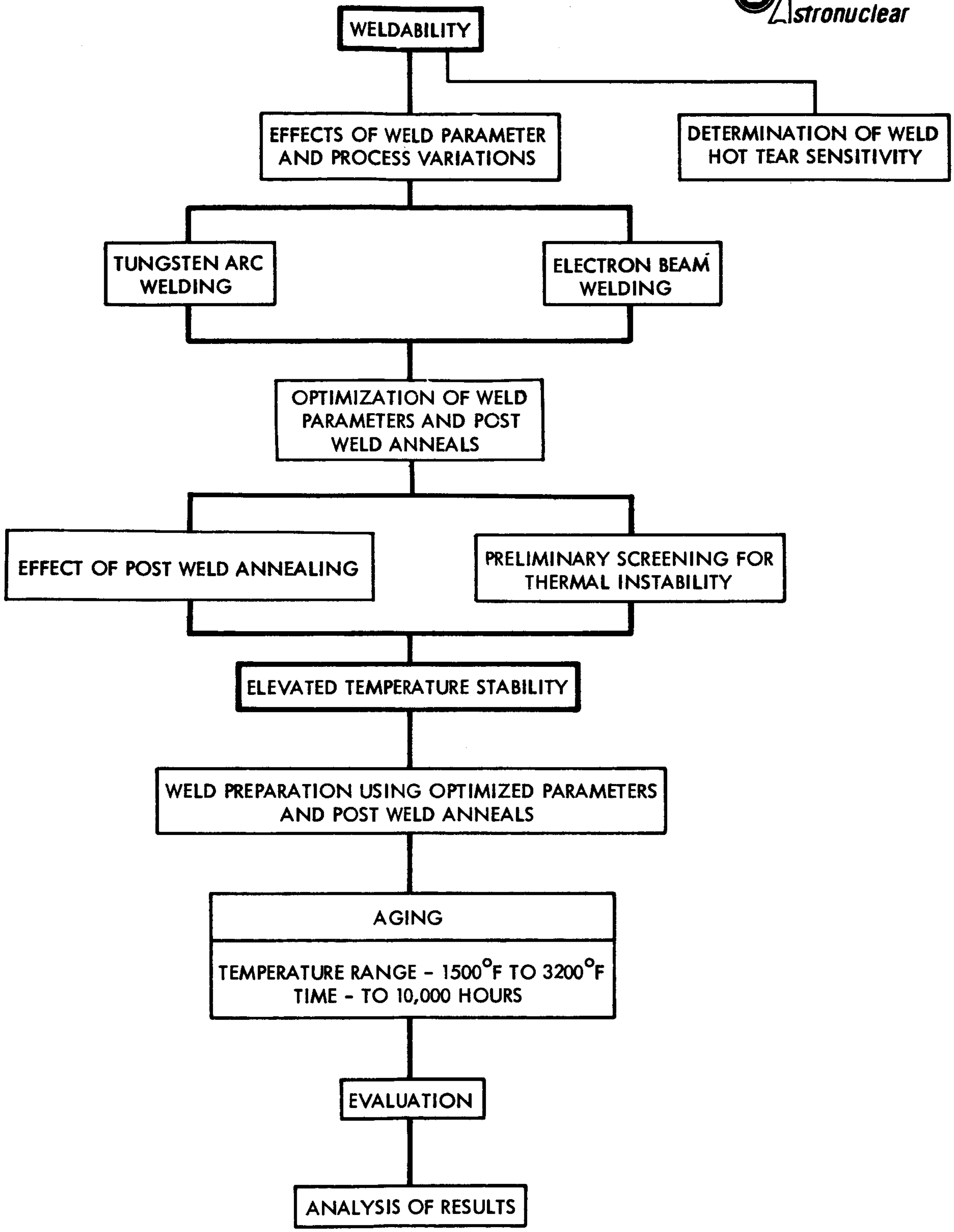


FIGURE 1 - Chronological Program Outline

TABLE I - Alloys Initially Selected for Weldability and Thermal Stability Evaluations

Alloy	Nominal Composition Weight Percent
AS-55	Cb-5W-1Zr-0.2Y-0.06C
B-66	Cb-5Mo-5V-1Zr
C-129Y	Cb-10W-10Hf+Y
Cb-752	Cb-10W-2.5Zr
D-43	Cb-10W-1Zr-0.1C
FS-85	Cb-27Ta-10W-1Zr
SCb-291	Cb-10W-10Ta
T-111	Ta-8W-2Hf
T-222	Ta-9.6W-2.4Hf-0.01C
Ta-10W	Ta-10W
W-25 Re	W-25 Re
W	Pure W

Note: All Alloys to be From Arc-Cast and/or Electron Beam Melted Material

II. SUMMARY

Procurement requests have been processed for all alloys except pure tungsten for which a pre-procurement evaluation was required. Delivery status is shown in Table II. Orders have been filled for three of the alloys. Because of the low production rates and unique character of most of these alloys, procurement, including specification preparation, is a considerable part of the total effort during the first phase of this program.

During the second quarterly period, a specification was prepared for the W-25 Re alloy and quotations were requested for both arc-cast sheet and powder metallurgy sheet. Arc-cast sheet has been ordered which, although more expensive, should prove to have better welding characteristics.

A pre-procurement evaluation of the weldability of pure tungsten resulted in the selection of arc-cast tungsten sheet for this program. Powder metallurgy sheet displayed a definite tendency for porosity formation in welds. Because of the heat to heat variability and unique handling problems involved in the welding and testing of tungsten, this evaluation will be limited in scope and directed primarily towards identification of any advantages in the use of arc-cast rather than powder metallurgy tungsten.

The AS-55 alloy was ordered following a negotiation delay resulting in this case from the fact that this is not a commercial alloy.

Arrangements were made to satisfy the sheet requirements for FS-85 by transfer of material produced on Contract NOw 63-0231-c for the Bureau of Naval Weapons by Fansteel Metallurgical Corporation.

The welding chamber atmosphere monitoring system was completed and placed into regular operation. This system provides for monitoring by two different oxygen gages and one moisture gage. Initial data indicate that an atmosphere of less than 5 ppm oxygen is easily obtained and has been shown to remain fairly constant throughout a typical weld run. A system response test demonstrated that the monitoring system responds to a sudden change in atmosphere oxygen level within fifteen seconds. A systematic study of total impurity levels, as measured by sampling and subsequent mass spectrometer analysis, was also undertaken.

Weld chamber tooling is being installed and great care is being exercised to identify the source of any weld atmosphere contamination which may accompany this installation.

TABLE II - Alloy Procurement and Delivery Schedule

Alloy	NTPB Approval	Req-For Quote	Quote Rec'd	Order Placed	Promised Shipping Date	Actual Delivery			Supplier
						Sheet	Plate	Wire	
AS-55	8/12	9/9	10/1	1/29/64	5/1/64				General Elec. (Cleveland)
B-66	8/12	7/26	8/19	8/29	10/18			11/8	Westinghouse
C-129Y	8/12	9/9	9/20	10/2	11/30	12/24			Wah Chang
Cb-752	8/12	9/9	9/19	10/21	11/30	12/31	12/18	12/31	Haynes
D-43	8/12	7/26	8/17	9/3	11/8	11/15	10/18		DuPont
FS-85	8/12	7/26	8/12	8/22	1/30/64		1/6		Fansteel
SCb-291	8/12	9/9	9/17	10/2	11/30	1/9	1/8	12/6	Stauffer
T-111	8/12	7/26	8/16	9/27	10/28		12/31	12/24	NRC
T-222		9/9	9/25	10/21	1/15/64				Westinghouse
Ta-10W	8/12	7/26	8/12	8/22	9/30	10/21	10/3	10/17	Fansteel
W-25 Re	8/12	11/8	11/26	2/10/64	4/1/64				Wah Chang
W		2/14/64							

The electron beam welder has been modified by the addition of a liquid nitrogen trap to improve vacuum capability following initial testing.

Quotations for ultra-high vacuum annealing furnaces were obtained and an order was placed with Varian Associates, Inc. for the units required in this program. These furnaces will be equipped with bakeout shrouds, adsorption pumps for roughing, and sputter-ion pumps for high vacuum holding.

Welding studies were initiated by producing weld restraint bead-on-plate patch tests in sheet from five alloys (Ta-10W, D-43, Cb-752, SCb-291, and C-129Y) and a circular groove restraint weld test in Ta-10W plate. In addition a weld joint configuration evaluation was undertaken for the 3/8 inch plate thickness being evaluated in this study. All welding tests were accompanied by continuous weld atmosphere monitoring.

III. TECHNICAL PROGRAM

A. ALLOY PROCUREMENT AND EVALUATION

The procurement status of the alloys included in this program is shown in Table II. All alloys except pure tungsten have been ordered. Tungsten procurement was delayed pending the outcome of a weld evaluation of sheet samples furnished by several suppliers. This evaluation, as described later in this report, is now complete and an order for tungsten sheet will be placed in the next period. Both the W-25 Re alloy and pure tungsten will be evaluated only in sheet form. Other alloys will be evaluated both as sheet and plate.

At the direction of the Bureau of Naval Weapons, FS-85 sheet produced under Contract NOw 63-0231-c is being furnished by the Navy for this evaluation. Under the Navy contract, FS-85 was converted to 0.050 inch sheet on a developmental basis by the Fansteel Metallurgical Corp. Hence, complete documentation, including the processing history of this material, will be available for reference in this evaluation.

Complete shipments have been received for three alloys (Cb-752, SCb-291, and Ta-10W) while partial shipments have been received on five other alloys. Material now available permits initiation of welding studies on six of the twelve alloys.

Special difficulty was encountered in ordering AS-55, which is not a commercial alloy, and W-25 Re, which has not yet been produced as sheet in significant commercial quantities. The AS-55 is being provided by the Lamp Metals and Components Department of General Electric on a special order basis. The W-25 Re alloy is being procured from the Wah Chang Corp. and will be furnished as sheet converted from arc-cast ingots. The W-25 Re alloy specification is included in Appendix I of this report. Specifications for the other alloys, except pure tungsten, were included in the First Quarterly Report.[1]

At the request of the NASA technical manager and with the concurrence of the TAPCO Materials Technology Division, sufficient additional W-25 Re sheet was ordered to satisfy the requirements of the NASA creep test program being conducted by TAPCO under Contract NAS 3-2545. Hence, material evaluated in both programs will be from the same heat and have the same processing history. This is desirable, particularly because of the limited data available on arc-cast W-25 Re.

Photomicrographs of the as-received material are shown in Figures 2 through 7. A completely recrystallized structure was observed for all

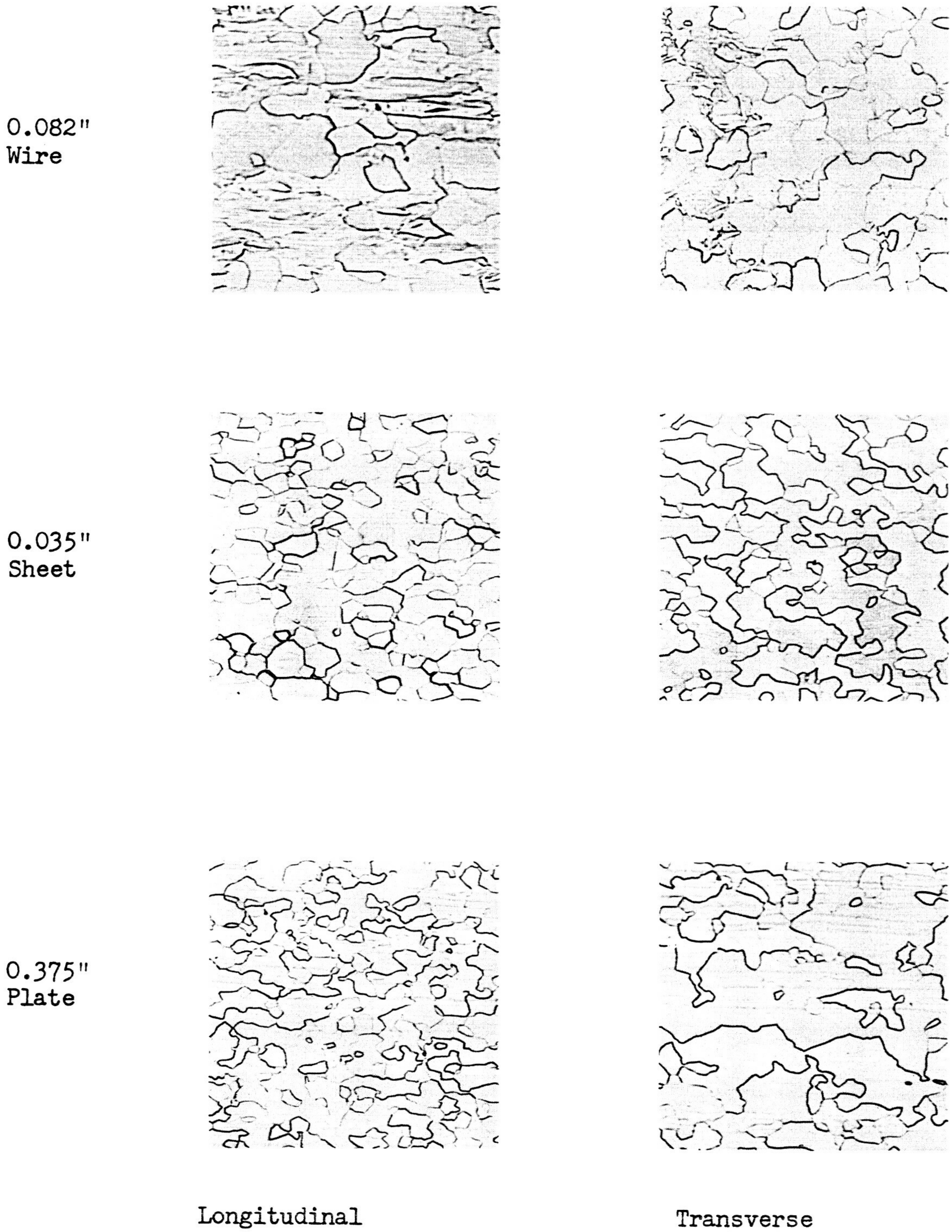
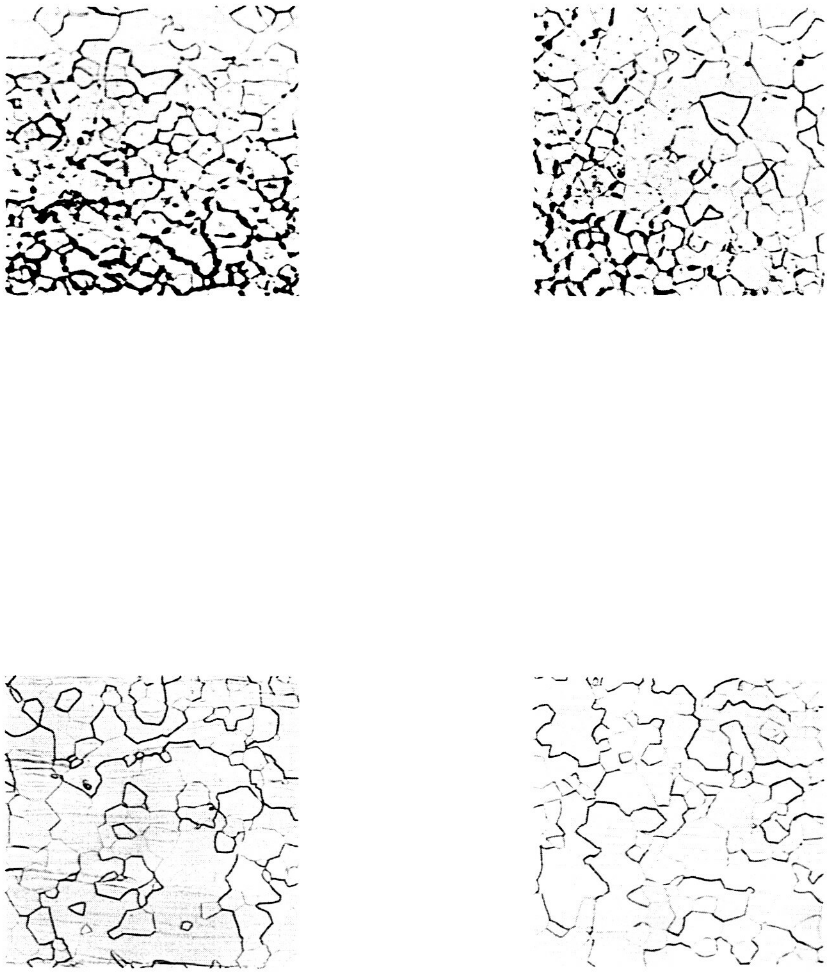


FIGURE 2 - Microstructures of As-Received Ta-10W, 100X
(HNO_3-$NH_4F.HF$ Etch)

0.082"
Wire

0.375"
Plate



Longitudinal

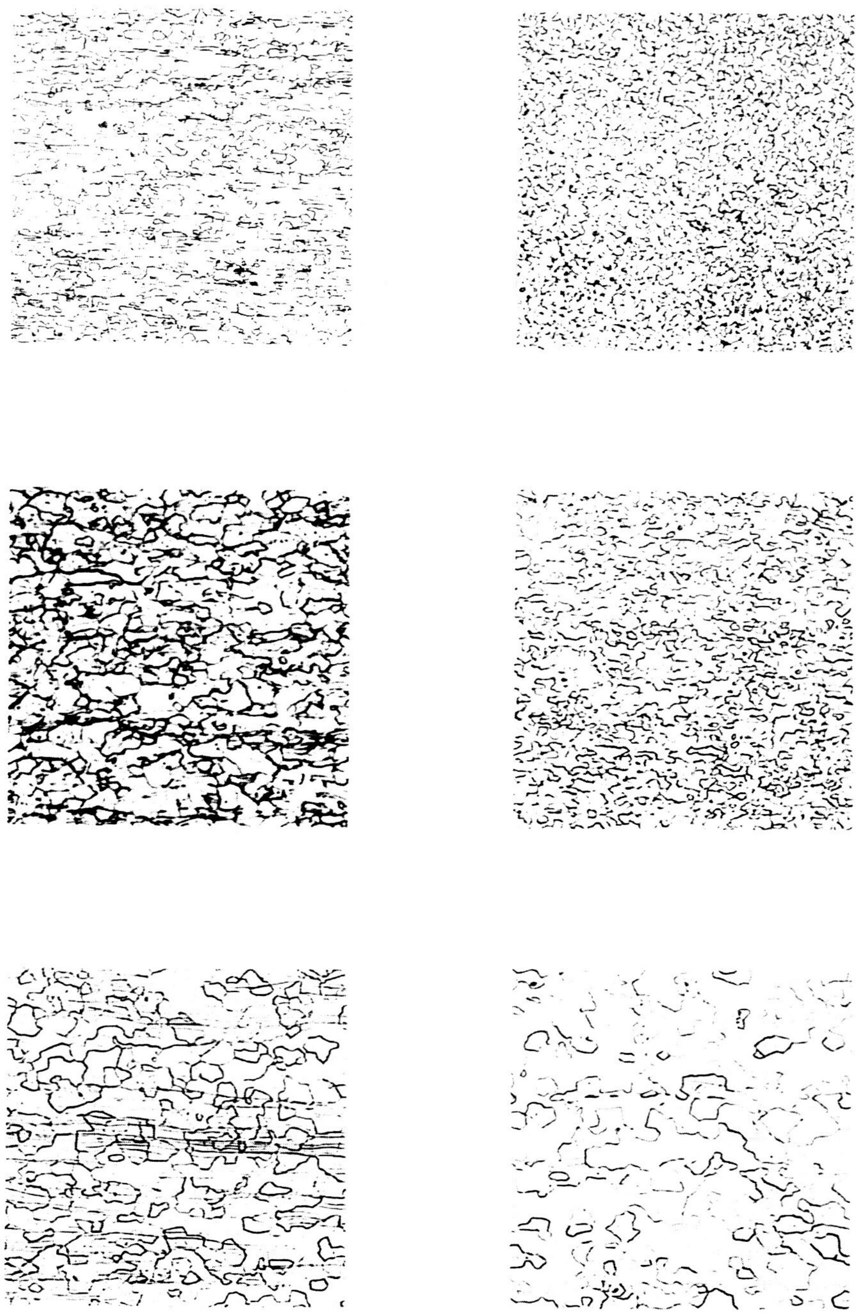
Transverse

FIGURE 3 - Microstructures of As-Received T-111, 100X
(HNO_3-$NH_4F.HF$ Etch)

0.082"
Wire

0.035"
Sheet

0.375"
Plate



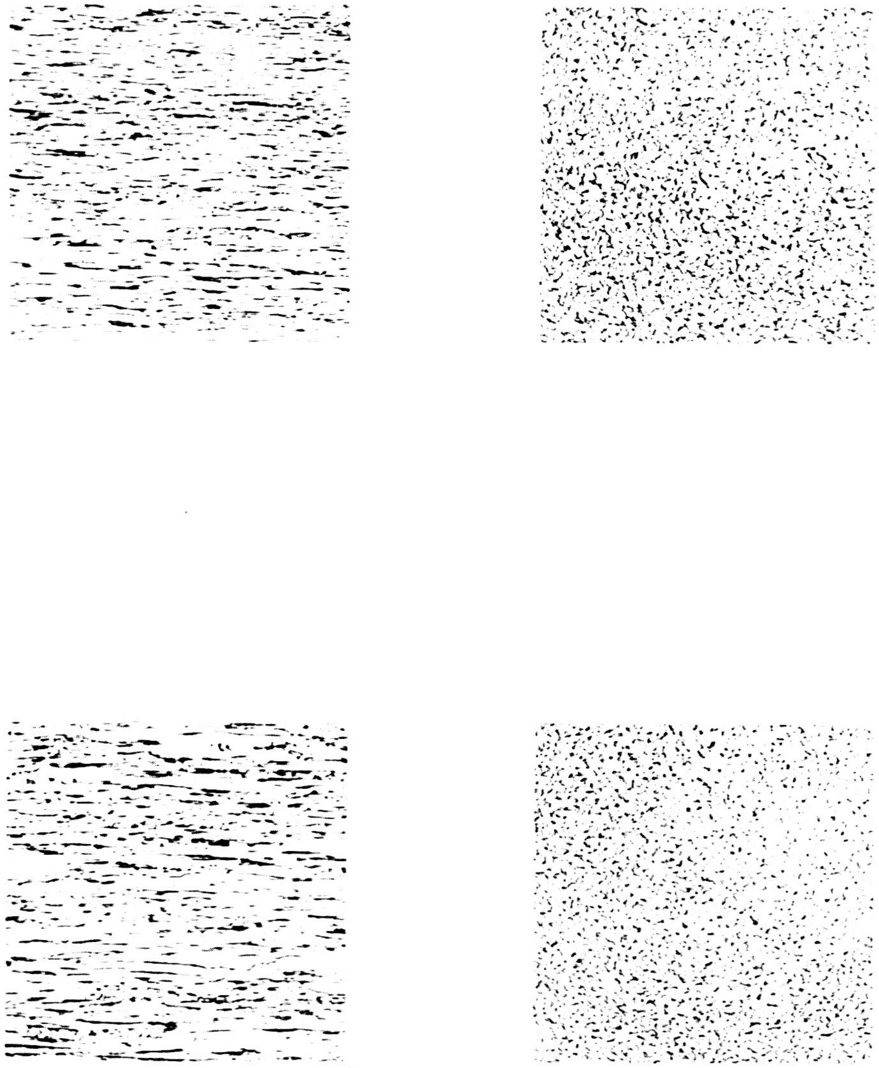
Longitudinal

Transverse

FIGURE 4 - Microstructures of As-Received Cb-752, 100X
(HNO_3-$NH_4F.HF$ Etch)

Heat W-569

Heat W-603

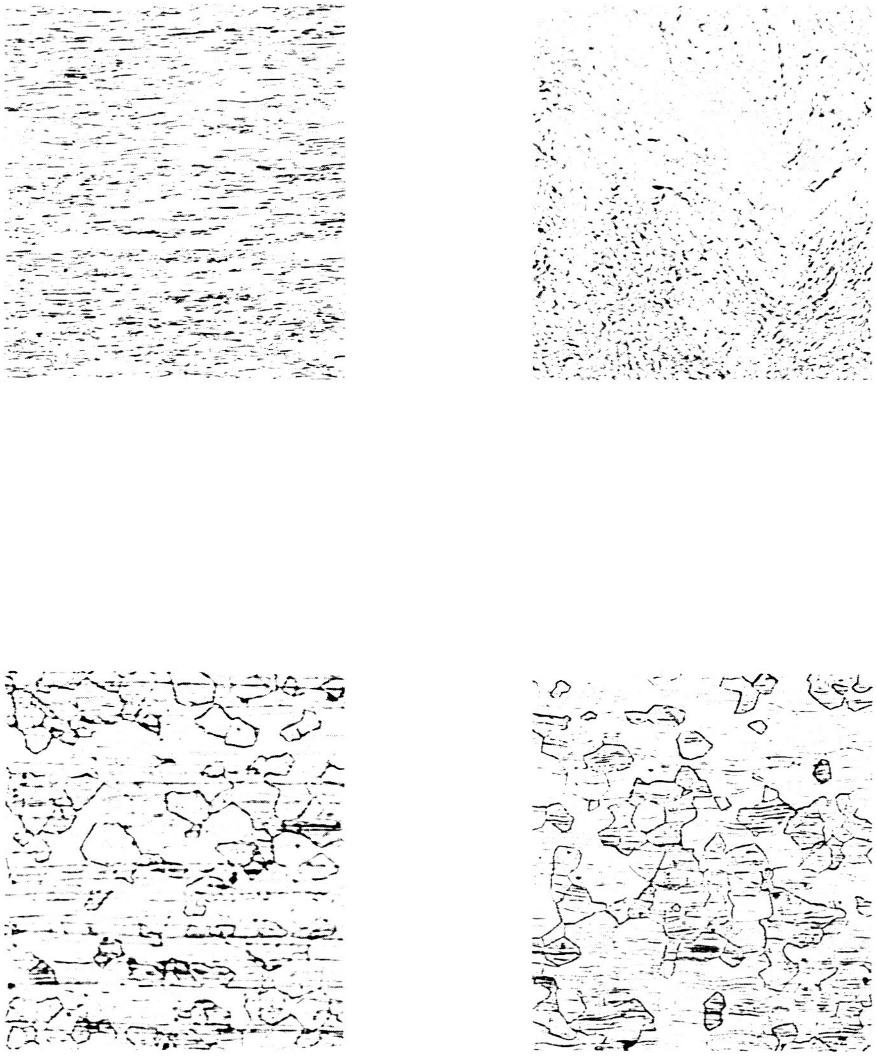


Longitudinal | Transverse

FIGURE 5 - Microstructures of As-Received B-66 0.082" Wire, 100X (HNO_3-$NH_4F.HF$ Etch)

0.082"
Wire

0.035"
Sheet



Longitudinal   Transverse

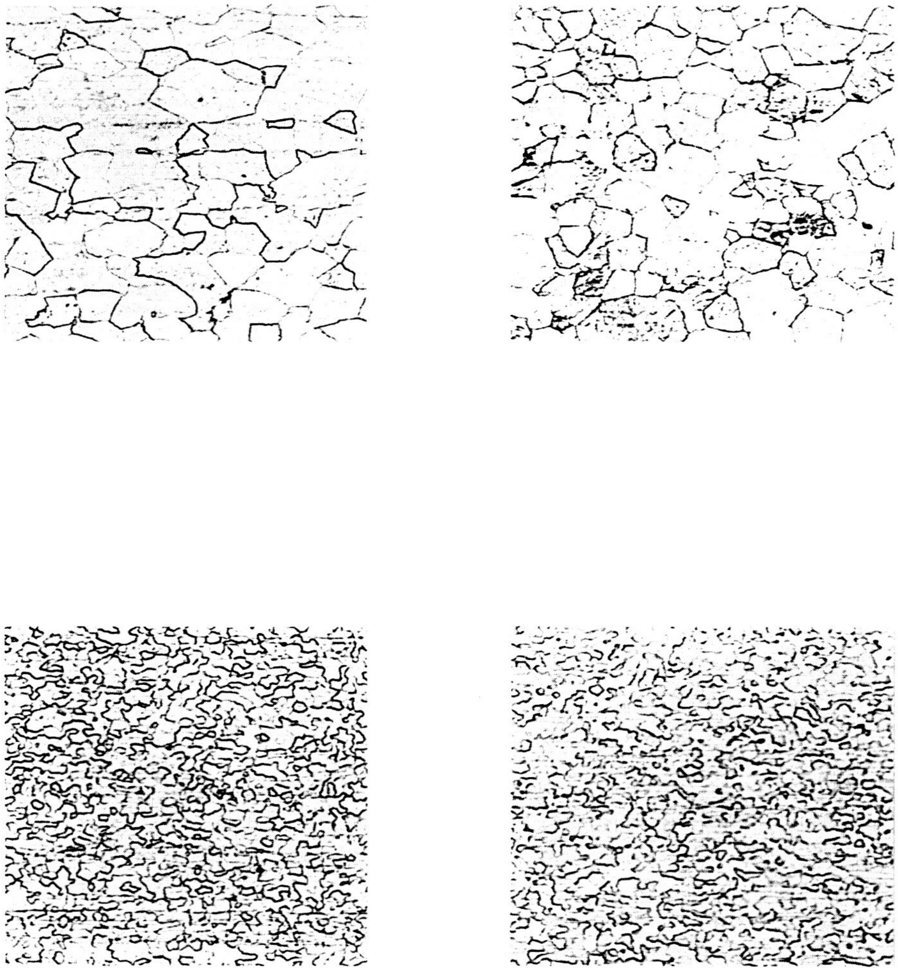
FIGURE 6 - Microstructures of As-Received SCb-291, 100X
(HNO_3-$NH_4F.HF$ Etch)

D-43

0.035"
Sheet

C-129Y

0.035"
Sheet



Longitudinal Transverse

FIGURE 7 - Microstructures of As-Received D-43 and C-129Y, 100X
(HNO_3-$NH_4F.HF$ Etch)

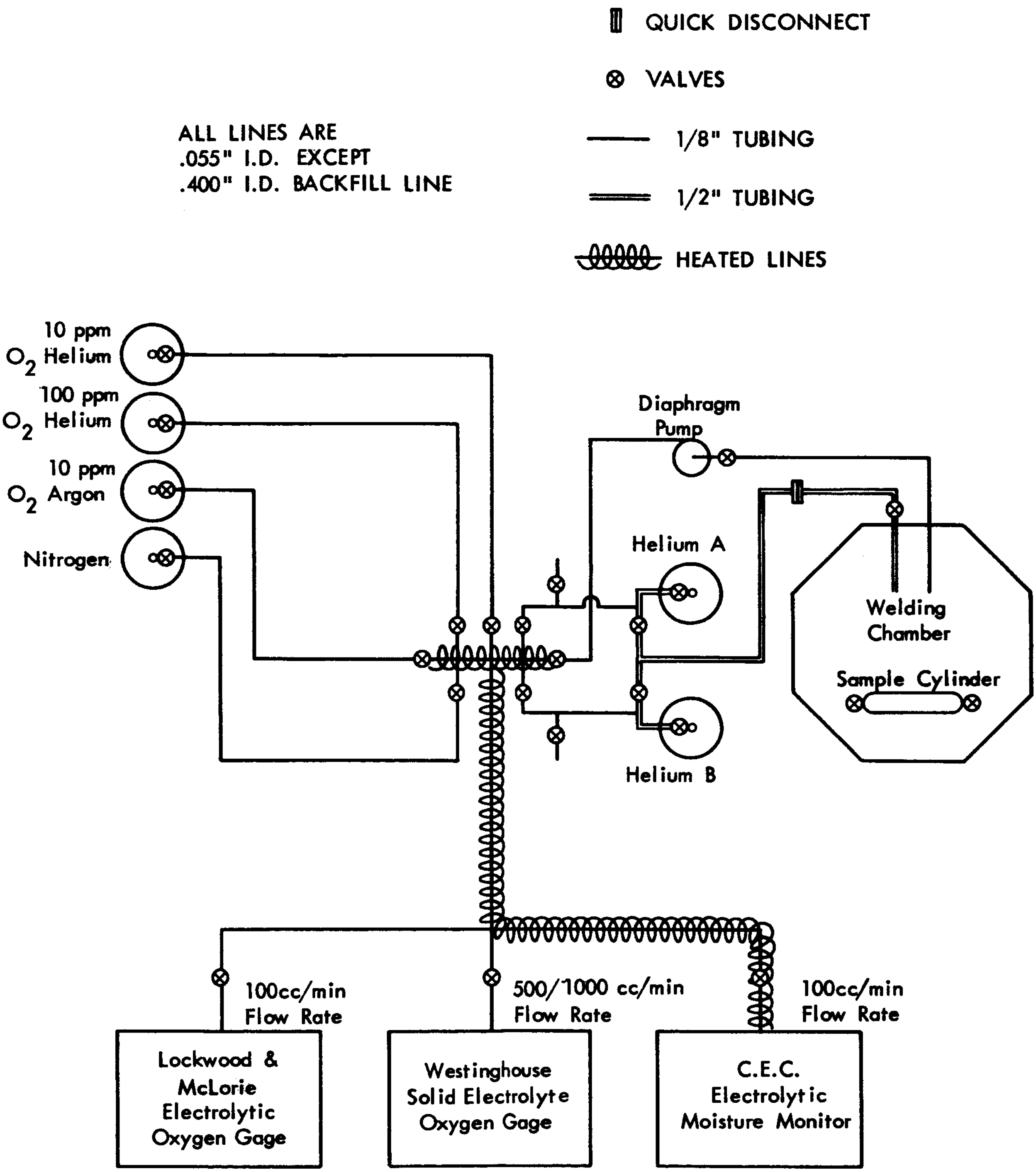


FIGURE 8 - Schematic of Gas Sampling and Monitoring System

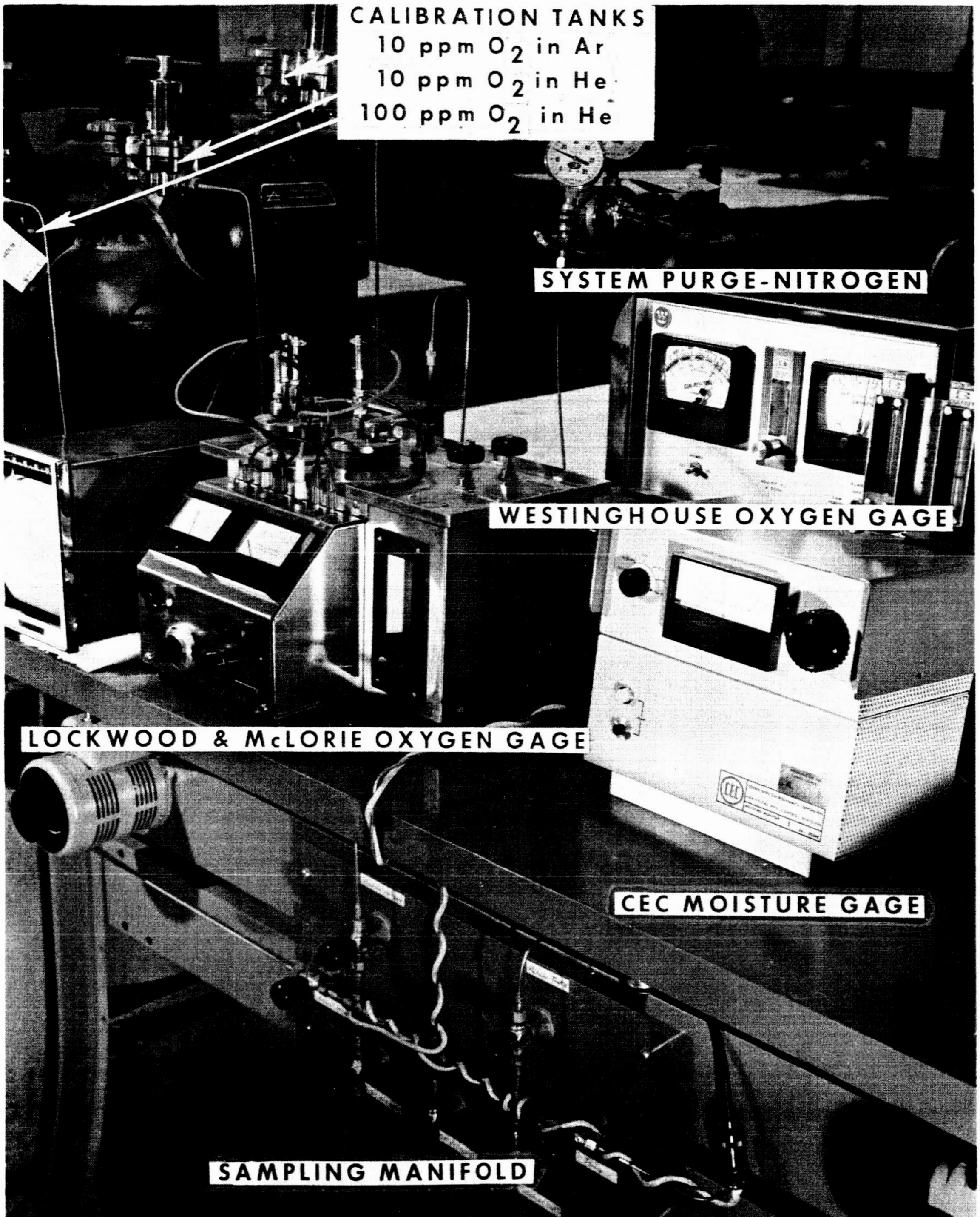


FIGURE 9 - Photograph of Atmosphere Monitoring Equipment

sheet and plate material except D-43 plate, which was received in the stress relieved condition. The T-111 wire, Figure 3, has apparently been contaminated during fabrication as indicated by the inhibited grain growth and grain boundary contamination of the wire surface. Over 50% of the wire diameter appears to have been affected. Appropriate action will be taken to obtain material meeting the requirements of the program.

B. WELD ATMOSPHERE MONITORING

The atmosphere monitoring system has been installed and operated successfully. Several minor changes designed to improve performance have been made including the installation of heating tape on the sampling lines to provide a convenient line bakeout system. A revised plan of the system is shown in Figure 8 and the system configuration is illustrated in Figure 9. Improved sealing of fittings has been achieved and the C.E.C. Moisture Monitor has been modified to eliminate an apparent water vapor trap in the flowmeter.

A Westinghouse solid electrolyte oxygen gage is now included in the atmosphere monitoring system. In addition to this gage, the system includes a Lockwood & McLorie liquid electrolyte oxygen gage and a C.E.C. Moisture Monitor. The oxygen gages are complimentary since the Westinghouse gage has a very fast response, but is sensitive to a combustible contaminant background gas, while the Lockwood & McLorie gage is relatively insensitive to background contaminants, but has a significantly slower response rate. Initial observations indicate that welding apparently releases a burst of combustible contaminants which produce a lower reading on the Westinghouse oxygen gage.

1. System Accuracy

A continual improvement in system integrity has been observed as shown in Table III, which lists the measured helium bottle impurity analyses chronologically. The high moisture levels recorded for helium tank numbers 25 and 26 were the result of incomplete sample line bakeout which disturbed but did not completely remove the tramp moisture in the upstream line. Later measurements reflect a more complete sampling system purge. Table IV, a list of the backfilled chamber analyses, also shows the same trend. To check the system accuracy, a 500 cc gas sample was drawn from the welding chamber as an oxygen and water vapor analysis was obtained with the atmosphere monitoring system. A comparison of the oxygen and water vapor readings of the monitoring system and a mass spectrometer total impurity analysis of the sample gas is shown in Table V. The mass spectrometer analysis was made at the Westinghouse Research Laboratories utilizing

TABLE III - Chronological Results of Helium Gas Bottle Analyses

Oxygen and Moisture Reading (ppm by Volume)					
Helium Bottle Number	Westinghouse O_2 Gage	Lockwood & McLorie O_2 Gage	C.E.C. Moisture Gage	Purge Time(1) Minutes	Test Date
37	1.5	2.4	6.4	120	1/15/64
36	4.0	5.6	9.5	60	1/15/64
35	1.2	2.2	4.2	220	1/7/64
34	3.5	4.8	N.A.	N.A.	12/27/63
33	1.5	1.9	9.0	120	1/8/64
32	N.A.	N.A.	N.A.	N.A.	N.A.
31	2.0	1.5	15.0	1200	12/13/63
30	N.A.	3.0	23.0	300	11/27/63
29	N.A.	3.9	23.0	960	11/26/63
28	N.A.	3.5	40.0	3600	11/23/63
27	N.A.	2.7	48.0	130	11/22/63
26	N.A.	4.7	230.0(2)	120	11/20/63
25	N.A.	3.0	240.0(2)	120	11/19/63
24	N.A.	3.2	46.0	1200	11/19/63

(1) Total Time Gas Bottle Was Connected To Gas Analysis System

(2) High Moisture Readings Resulting From Incomplete Sample Line Bakeout

N.A. Indicates: Not Available

TABLE IV - Typical Weld Box Atmosphere Readings

Oxygen and Moisture Reading (ppm by Volume)					Box Diameter
Test Date	Westinghouse O_2 Gage	Lockwood & McLorie O_2 Gage	C.E.C. Moisture Gage	Helium Bottle Number	
1/15/64	3.0	4.8	10.0	37	42" **
1/14/64	8.0	6.0	20.0	N.A.	42"
1/9/64	1.0	N.A.	22.0	N.A.	42"
1/7/64	N.A.	2.7	24.0	35	42"
1/6/64	4.5	4.8	15.0	35	42"
12/31/63	N.A.	3.8	N.A.	34	42"
12/30/63	N.A.	2.6	N.A.	34	42"
12/30/63	1.0	5.0	N.A.	34	42" *
12/27/63	1.0	6.5	N.A.	34	42"
12/4/63	N.A.	8.0	N.A.	N.A.	18"
12/2/63	N.A.	5.0	15.6	30	18"
11/26/63	N.A.	30.0	12.0	29	18"
11/25/63	N.A.	8.5	27.4	N.A.	18"
11/22/63	N.A.	75.0	87.0	N.A.	18"

** Sample Bottle Analyzed By Mass Spectrometer

* Weld Chamber Idle Overnight

N.A. Indicates: Not Available

TABLE V - Comparison of Atmosphere Analysis Methods

Analysis Method		ppm (by volume)
Bottle Analysis at WANL[1]	Westinghouse O_2 Gage	1.5 O_2
	Lockwood & McLorie O_2 Gage	2.4 O_2
	C.E.C. Moisture Monitor	6.4 H_2O
Backfilled Chamber Analysis	Westinghouse O_2 Gage	3.0 O_2
	Lockwood & McLorie O_2 Gage	4.8 O_2
	C.E.C. Moisture Monitor	10.0 H_2O
Mass Spectrograph Analysis[2]	Neon	7.3
	Chlorinated Hydrocarbon	6.7
	Nitrogen	3.4
	Oxygen	1.2
	Argon	0.9
	Hydrogen	0.6
	Carbon Dioxide	0.1
	Water	<5.0
	Total Measurable Impurities	20.2

(1) All Helium Gas From Bottle No. 37

(2) Sample Obtained From Backfilled Chamber

cryogenic concentration of impurities to produce a very sensitive analysis from a comparatively small gas sample[2] (500 cc). The concentrated impurities were then analyzed by a C.E.C. Model 21103C analytical mass spectrometer. The mass spectrometer analyzed moisture content was less than the minimum detectable level of 5 ppm obtainable with the sample size and sampling procedure used. The low sensitivity to water vapor is due to the adsorption of water vapor on the previously evacuated interior surfaces of the mass spectrometer and the sample bottle.[3] A means of obtaining equilibrium adsorption-desorption conditions in the sample bottle will be investigated in future analyses.

A chlorinated hydrocarbon was found to be a major impurity of the sample cylinder analysis. Future mass spectrometer analyses will include techniques designed to identify the source of contamination and will include an analysis of a helium tank and a welding chamber backfill of the same tank. Similar techniques will be used to identify the combustible impurity released by the welding operation.

2. Calibrated Gas Analysis

The unique chamber monitoring and gas sampling system provides a means of rapidly introducing a standard gas to the analysis system. Figure 10 shows the response of the Lockwood & McLorie oxygen gage as the sampling system is valved from a standard bottle of helium to a calibrated bottle containing 10.3 ppm oxygen. The small peak occurring when the sampling system was switched to the regular bottle of helium is due to pressure induced flow variations in the Lockwood & McLorie gage. Figure 11 represents the chamber monitoring system performance with the Lockwood & McLorie gage used for oxygen analysis. The initial high oxygen indication is due to residual contamination of the idle sampling line. No immediate change in oxygen level was observed during the welding operation.

3. Water Vapor Delay

Since the analysis system was placed in operation, an annoying delay was noticed in the response of the moisture monitor to changing moisture levels. In an effort to determine the real capabilities of the moisture analyzing system, the C.E.C. Moisture Monitor was removed from the gas sampling system and connected directly to a helium bottle as shown in Figure 12. A Linde R-5100 stainless steel gas regulator with a stainless steel diaphragm was used to avoid moisture pickup from the rubber-fabric diaphragm commonly used for helium gas regulators. "Wrap around" heater cords were used to bakeout the sample line to about (200°F) to reduce the system purge time. The rotometer was found to be a moisture trap

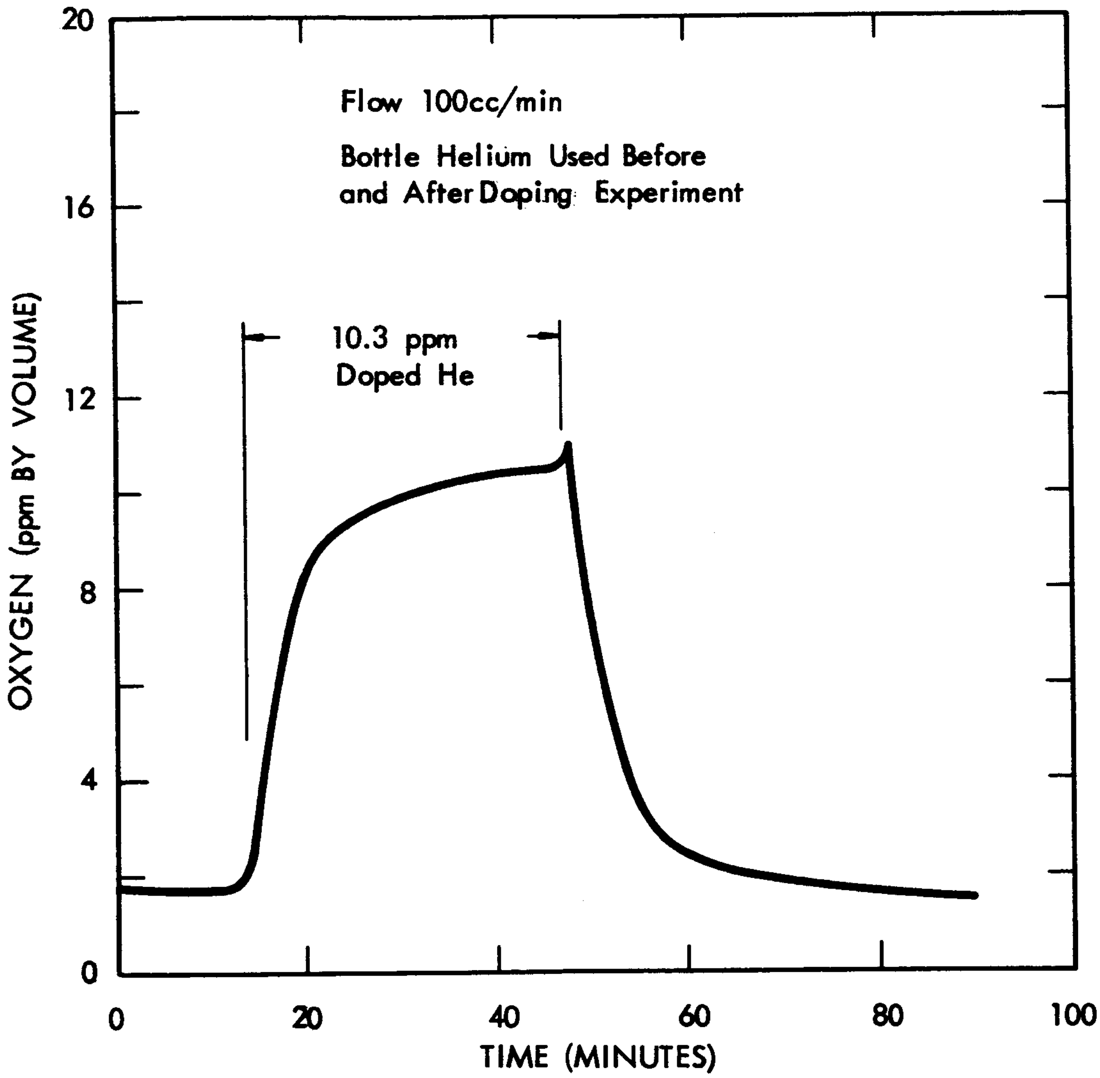


FIGURE 10 - Response of Lockwood and McLorie Oxygen Gage to Oxygen Doped Helium

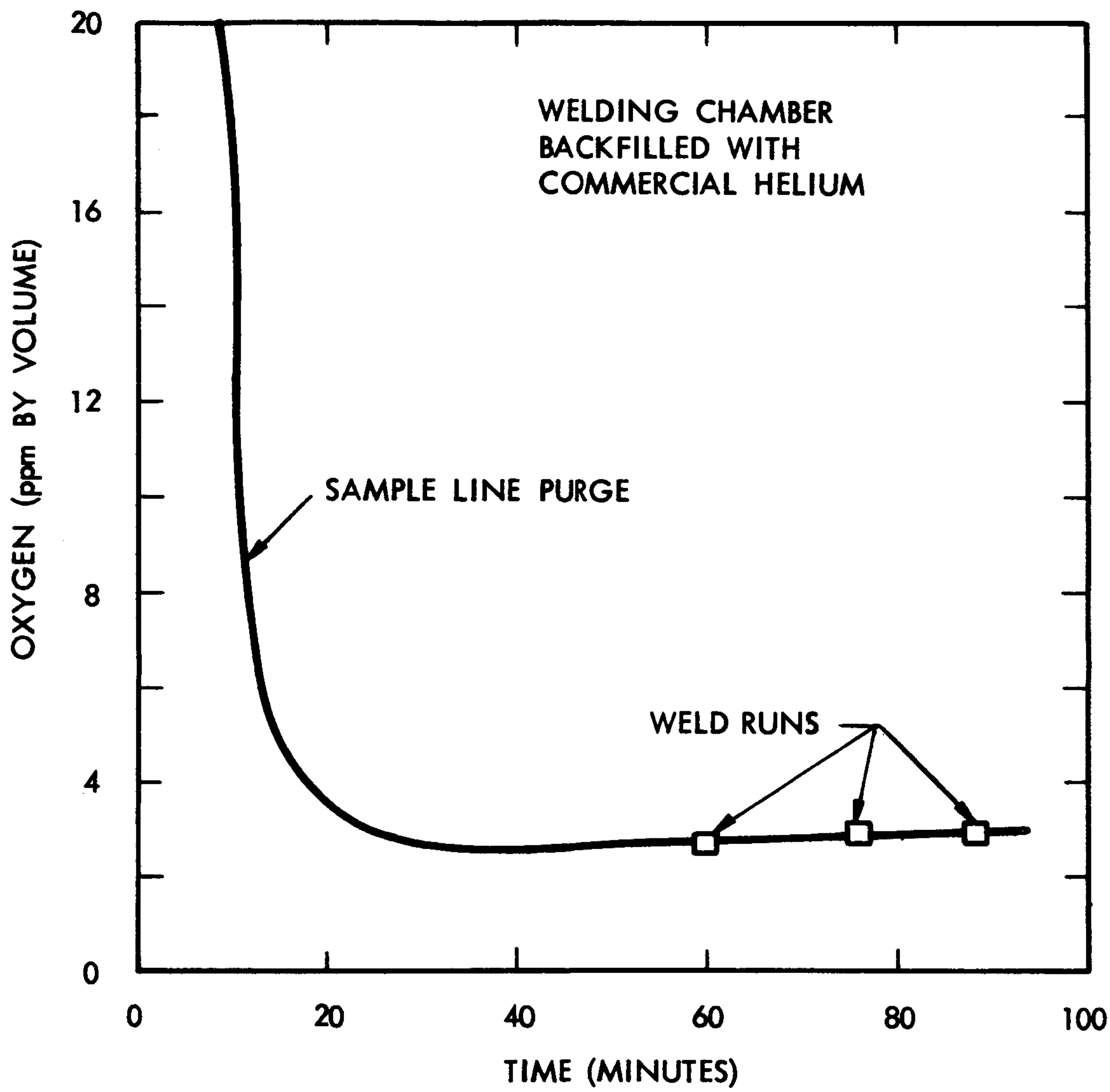
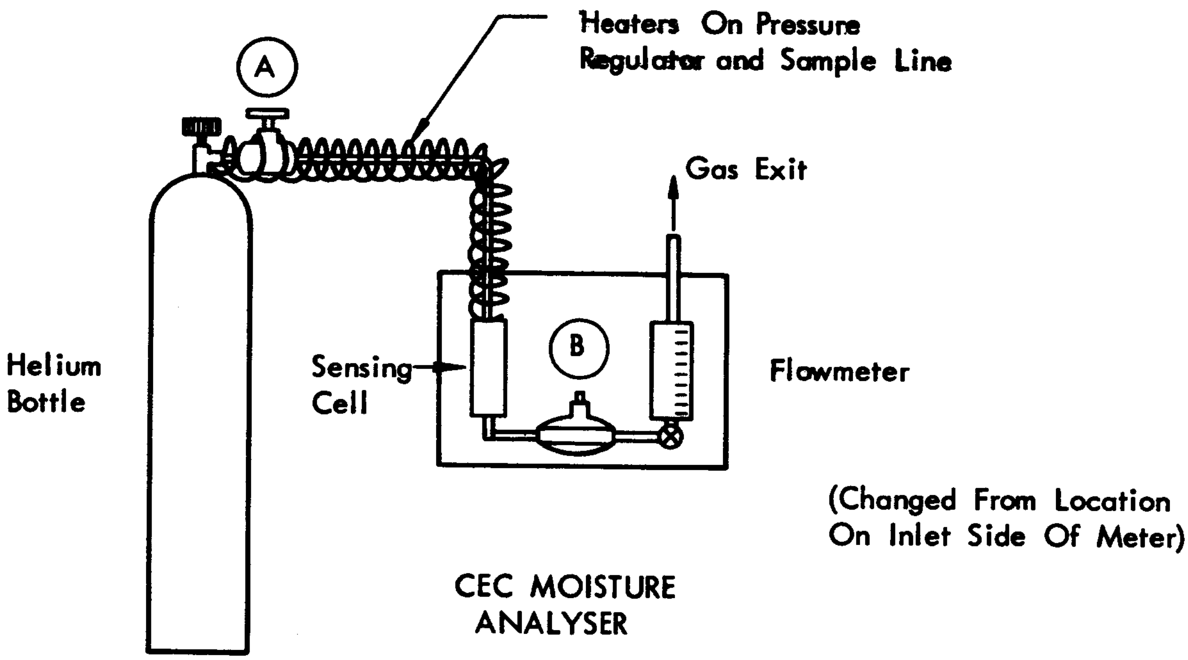


FIGURE 11 - Recorded Oxygen Level of Chamber Atmosphere During Welding



(A) Linde Series R-5100
Stainless Steel Pressure Regulator

(B) Flow Regulator
40" of 1/8" Stainless Steel
Tubing Used For Supply Line

FIGURE 12 - Schematic of Moisture Analyser Performance Test

and the C.E.C. gage was modified to place the rotometer downstream from the moisture sensing cell. With this improvement, a reading of 3 ppm moisture was obtained at 100 cc/min. flow for 16 hours without a sample line bakeout.

4. System Response to Introduced Impurities

To determine the response capabilities of the atmosphere monitoring system to a sudden leak, an experiment was devised to quickly add a measured amount of oxygen and water vapor to a normal high purity welding chamber atmosphere. A five pint glass bottle containing air and a measured amount of water was placed in the welding chamber and the chamber was normally evacuated and backfilled with helium. The level of oxygen and water vapor in the bottle was 200,000 ppm which was diluted by the large chamber volume to 360 ppm. (See Appendix II for calculations). Figure 13 shows the response of the monitoring equipment to the suddenly increased impurity level. Within one minute after the impurity release, (by breaking the bottle) the Westinghouse gage had reached 2/3 of the equilibrium level. The fast response of the Westinghouse gage is partly attributable to the high sample flow rate used which was 500 cc/min. compared to 100 cc/min. for both the Lockwood & McLorie oxygen gage and the C.E.C. Moisture Monitor. All three gages, however, are supplied by a common 1/8 inch stainless steel supply line and diaphragm pump to within 3 feet of the individual meters so that all the gages benefited from the high flow rate of the Westinghouse gage. The even greater recommended 1000 cc/min. flow for the Westinghouse gage was not obtainable because of limited pumping capacity for the sampling system. The initial high peaks observed for both oxygen and moisture level were probably due to incomplete diffusion of the impurities throughout the welding chamber. The equilibrium levels of both oxygen and water vapor were in close agreement to the calculated values.

C. WELDING PROGRAM

1. Weld Hot Tear Sensitivity

The available alloys have been evaluated for weld hot tear sensitivity employing a bead-on-plate patch test for sheet material and a circular groove restraint test for plate material. These tests were described in the First Quarterly Report.[1] The circular groove restraint test is complete only for the Ta-10W alloy for which both plate and filler wire were available.

Bead-on-plate patch tests were made in five alloys. These were C-129Y, D-43, Ta-10W, SCb-291, and Cb-752. Photographs of weld

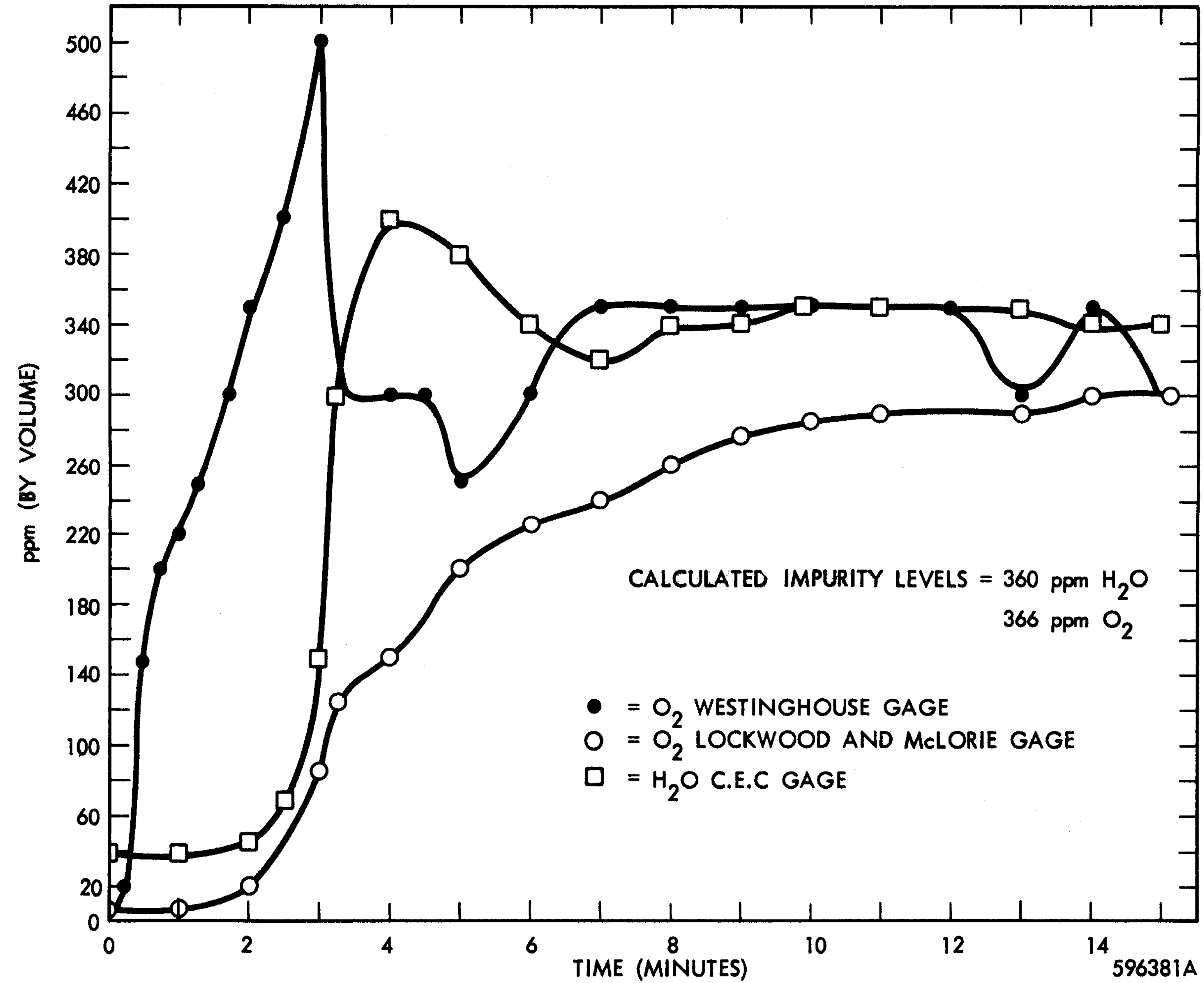


FIGURE 13 - Response of Weld Chamber Atmosphere Monitoring System to Introduced Contamination

restraint test specimens are shown in Figures 14 through 17. Specimens are shown both as-welded and as dye-penetrant inspected. Fairly severe stresses are induced in this test as indicated by warpage of the specimen. Welds were made manually by first producing the two-inch diameter circle and then the two cross welds. A welding current of 40 to 60 amperes direct current, straight polarity, was required for the patch tests, all of which were welded in helium. The oxygen contamination level during welding reached a high of 3.6 ppm after one hour of welding from an initial level of 2.7 ppm.

The circular groove restraint weld was produced by first running a fusion pass (no filler wire) along the bottom of the groove and then filling the groove with two successive weld passes. A tungsten electrode was used and filler metal was added manually. The fusion root pass was made at 340 amperes, direct current-straight polarity, while filler passes were made at 300 amperes. Monitoring of the helium welding atmosphere during this welding showed that the oxygen level remained less than 3 ppm.

The circular groove restraint test was evaluated by radiography as well as visual and dye-penetrant inspection. No defects were noted. Point indications noted on the dye-penetrant inspected patch test specimens occurred only at weld craters. These indications result from improper weld tailing (decay), and are not particularly indicative of hot tear sensitivity.

2. Tungsten Weldability

At the direction of the NASA technical manager, a limited pre-procurement screening of pure tungsten sheet for weldability was undertaken to assist in preparation of specifications and in identifying potential sources of supply. This effort was necessarily of limited scope.

Tungsten samples were obtained from seven potential suppliers. These were: General Electric, Fansteel, National Research Corporation (Distributor for Metallwerke Plansee), Wah Chang, Climax Molybdenum, Universal Cyclops, and Rembar Co., Inc. Five suppliers furnished powder metallurgy sheet while two suppliers furnished arc-cast tungsten sheet. From these samples, two sets of bead-on-plate welds were produced and one set of butt welds was made. All bead-on-plate welds were made manually using the TIG welding process and, because the sample thickness varied between 0.020 inch and 0.050 inch, welding current was varied accordingly. Weld evaluation was directed primarily towards determination of weld soundness, particularly as regards freedom from porosity, a common problem in tungsten weldments.

Welds were evaluated on the basis of visual, radiographic, and metallographic inspection. No mechanical evaluation was made. All welding was done without preheat, resulting in varying degrees of brittle fracturing during welding. The severity of this problem is clearly shown by the cracking displayed in the butt welded samples shown in Figure 18. This problem was minimized in the bead-on-plate welds by using narrow strips (3/4 inch wide) and supporting the specimen in a cantilever fashion during welding minimizing the weld cooling rate and providing, effectively, a considerable specimen preheat shortly after weld initiation.

In general the tendency toward cold cracking increased with thicker sheet and was less in the arc-cast material. All welds were extremely brittle and would fracture with very slight, low load, bending at room temperature. Radiographs highlighted the weld structure, possibly because of re-emmission during radiography. Porosity was not evident in the radiographs. Porosity was noted, however, in metallographic examination. Considerably more porosity was evident in the powder metallurgy sheet than in the arc-cast sheet as shown in Figures 19, 20, and 21. Porosity in the powder metallurgy sheet was most pronounced at the fusion zone-heat affected zone interface. Based on these results, arc-cast tungsten sheet will be procured for this program.

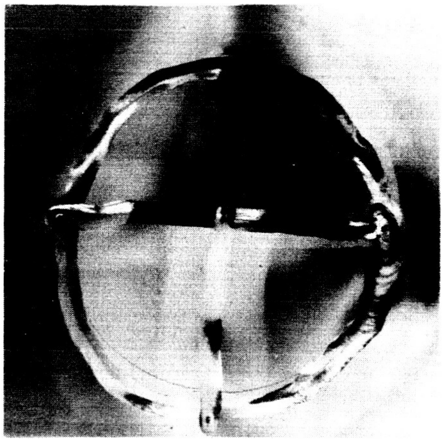
D. SPECIAL EQUIPMENT

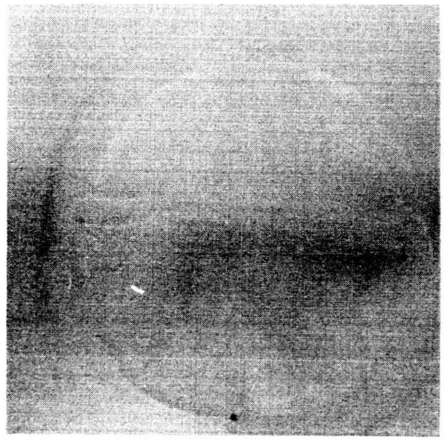
1. Weld Chamber Tooling

Weld chamber tooling has been fabricated and is being installed in the welding chamber. The welding fixtures are constructed of stainless steel and aluminum to provide corrosion resistant surfaces. All bearings are disassembled and degreased prior to installation. Installation is being undertaken in an orderly sequence with the weld chamber atmosphere being monitored at each step to insure that no tooling component proves detrimental to atmosphere quality. The work traverse table will provide a welding speed range of 4 to 60 ipm.

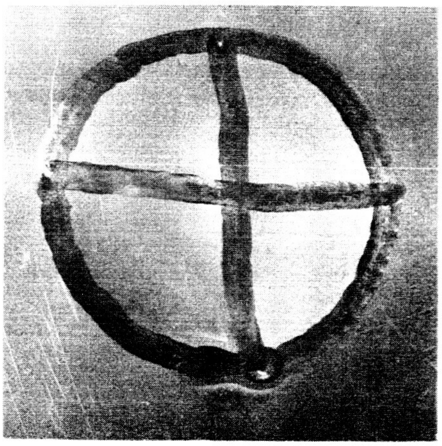
2. Electron Beam Welder

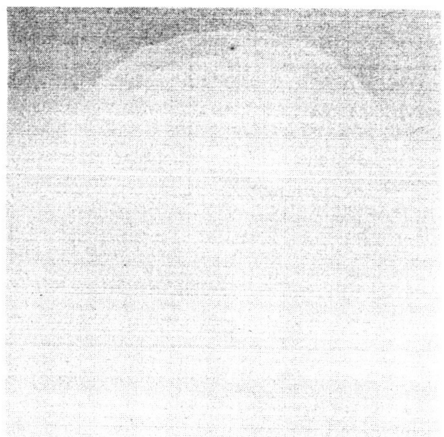
The Hamilton-Zeiss WO-2 electron beam welder was installed and checked out for vacuum performance. An ultimate vacuum of 2×10^{-5} Torr was obtained after a seven hour evacuation time. A CVC BC-60 chevron high vacuum liquid nitrogen baffle has been installed along with Viton-A "O" rings. The performance of the modified vacuum system will soon be evaluated with the electron beam welder in its permanent location.





Ta-10W



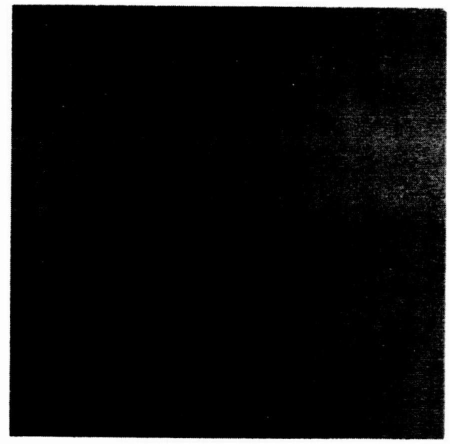


SCb-291

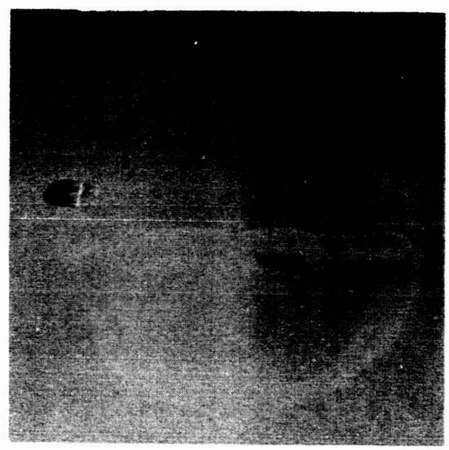
As-Welded

Dye Penetrant Inspected

FIGURE 14 - Photographs of Patch Test For 0.035" Sheet



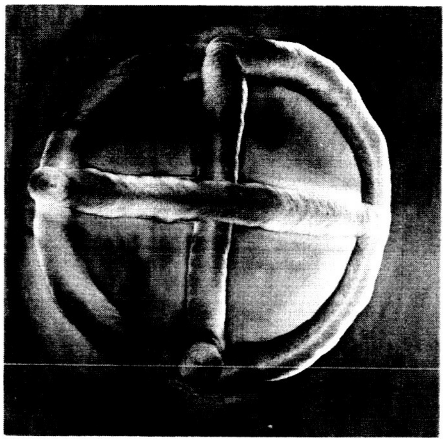
C-129Y

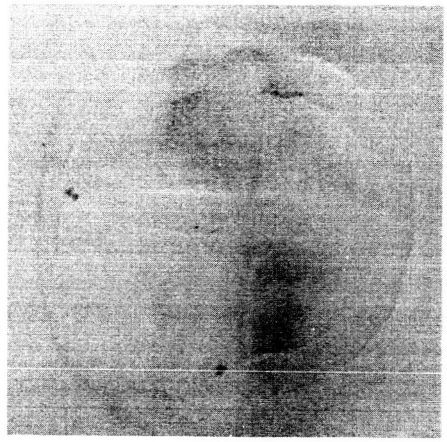


D-43

As-Welded | 1 2 INCHES | Dye Penetrant Inspected

FIGURE 15 - Photographs of Patch Test For 0.035" Sheet





Cb-752

As-Welded

Dye Penetrant Inspected

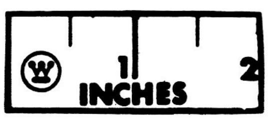
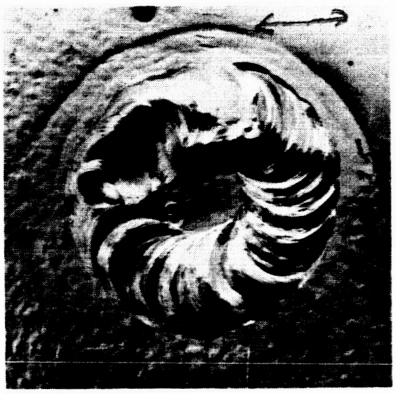
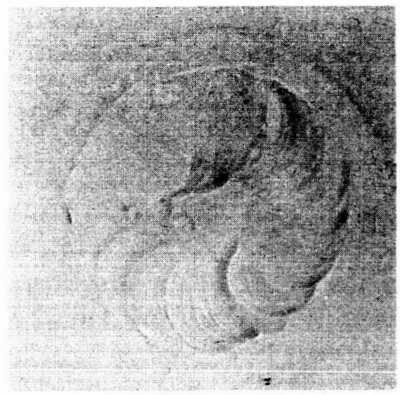


FIGURE 16 - Photographs of Patch Test For 0.035" Sheet



As-Welded



Dye Penetrant Inspected

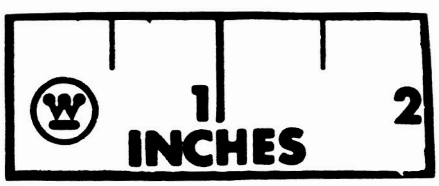
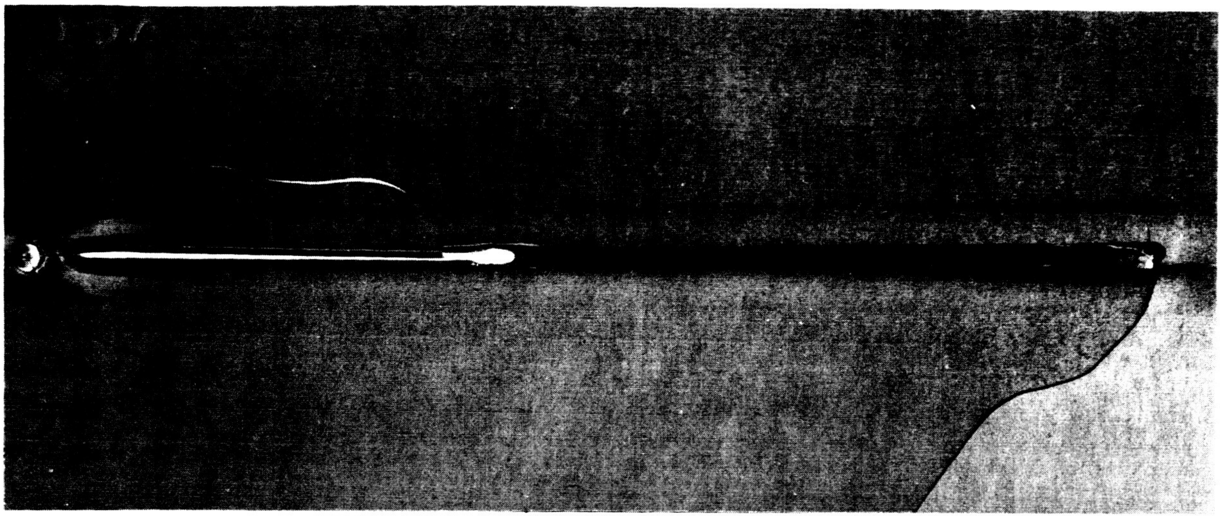
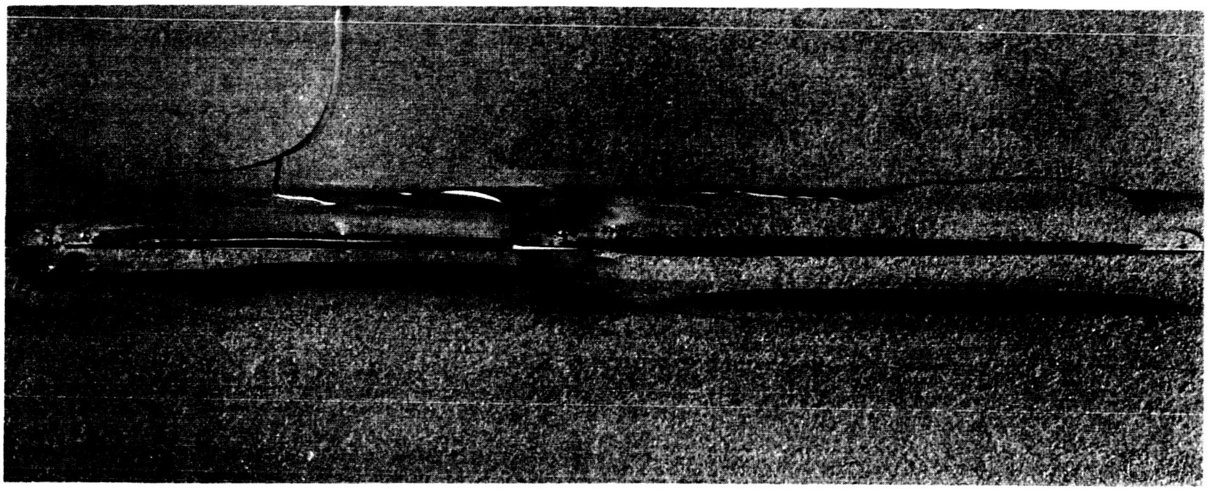


FIGURE 17 - Photograph of Circular Groove Weld Restraint Test in 3/8" Plate of Ta-10W Alloy



0.020 Inch Sheet



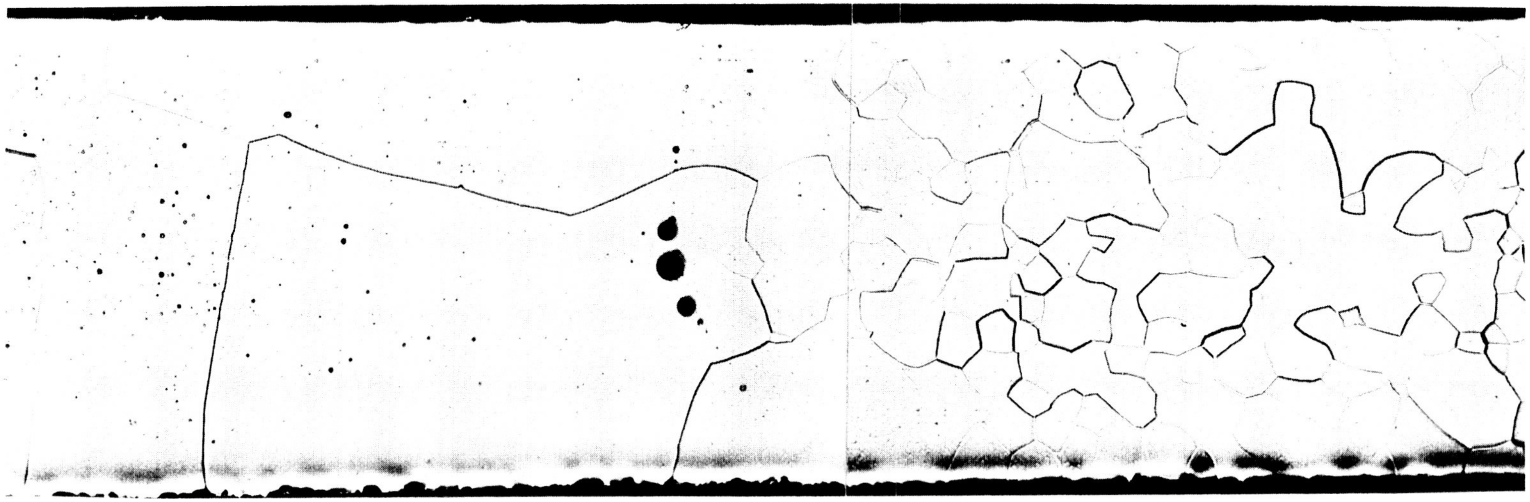
0.050 Inch Sheet

FIGURE 18 - Two Trial TIG Butt Welds in Arc-Cast Tungsten Sheet. Tightly Fixtured and Welded Without Preheat.

Supplier A - Arc Cast Tungsten 100X Neg. 2741A-B

Supplier B - Arc Cast Tungsten 50X Neg. 2742A-B

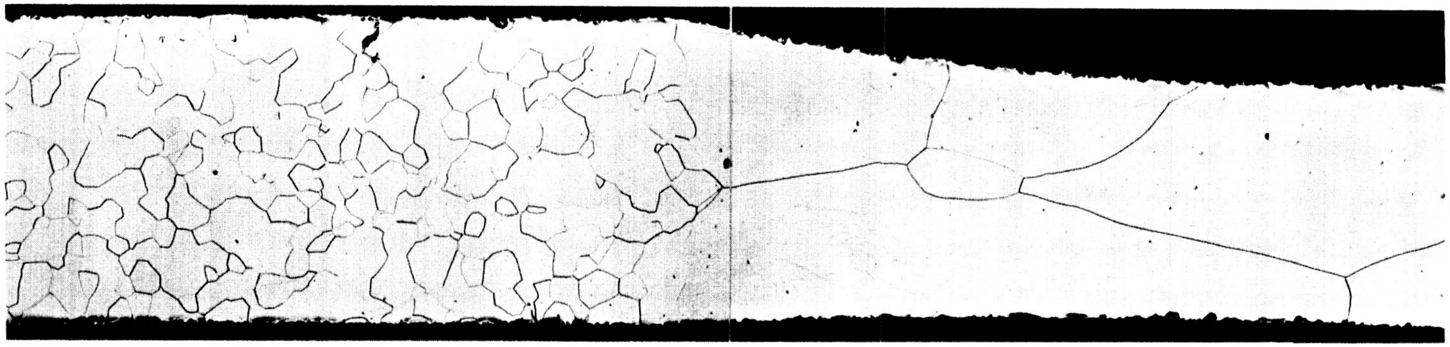
FIGURE 19 - Microstructures of Tungsten Welds (Electrolytic Etch NaOH)



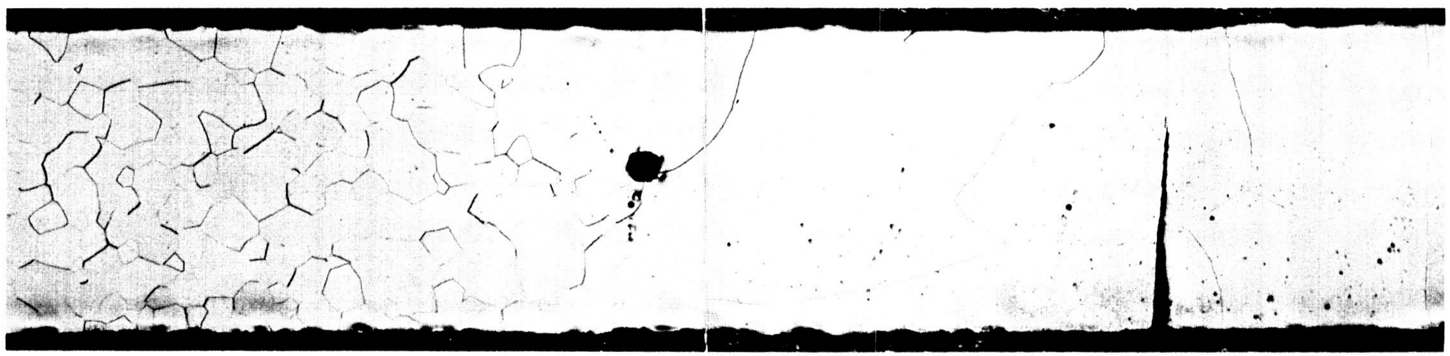
Supplier C - Powder Metallurgy Tungsten 100X Neg. 2738

Supplier D - Powder Metallurgy Tungsten 100X Neg. 3399

FIGURE 20 - Microstructures of Tungsten Welds (Electrolytic Etch NaOH)



Supplier E - Powder Metallurgy Tungsten 100X Neg. 2739



Supplier F - Powder Metallurgy Tungsten 100X Neg. 3318

FIGURE 21 - Microstructures of Tungsten Welds (Electrolytic Etch NaOH)

3. Ultra-High Vacuum Furnace

Manufacturing drawings have been received and approved for ultra-high vacuum furnaces allowing Varian Associates to begin manufacturing. Delivery of the first furnace is scheduled for March. A Varian Associate's creep testing furnace of similar heater and vacuum system design has been functionally tested to 10^{-9} Torr at 3500°F and the equipment has performed to satisfaction.

IV. FUTURE WORK

More extensive welding studies on both plate and sheet material will be initiated during the next period, particularly those necessary to firmly establish alloy base-line weldability data.

Evaluation of the weld atmosphere monitoring system will be continued with additional tests being run as dictated by experience gained during initial welding runs.

Early delivery of the aging furnaces and consequent installation and checkout is anticipated.

V. REFERENCES

1. G. G. Lessmann and D. R. Stoner, "Determination of the Weldability and Elevated Temperature Stability of Refractory Metal Alloys", First Quarterly Report WANL-PR-(P)-001

2. W. M. Hickam, Westinghouse Research Laboratories, Private Communication

APPENDIX A

Calculation for Added Impurity Level
(See Section III-B)

Calculation for Added Impurity

I. Ratio of Chamber Volume to Container Volume

Welding Chamber Volume (calculate from dimensions) = 82,800 $in.^3$
48 $ft.^3$
1,350 liters

5 pint container Ratio = $\frac{82,800}{144 \text{ in.}^3} = 573:1$

II. Oxygen Addition - Concentration When Diluted With Larger Box Volume

Air Composition is 210,000 ppm Oxygen

$$\frac{210,000 \text{ ppm}}{573} = 366 \text{ ppm oxygen}$$

III. Water Addition - 360 mg added in bottle as bulk addition
9 mg added as 40% relative humidity air
369 mg total water introduced

369 mg/1350 liters = 0.273 mg/liter

0.273 mg/ liter = 360 ppm water

APPENDIX B

Procurement Specification for Tungsten-25 w/o Rhenium Alloy Sheet

Astronuclear

Westinghouse Electric Corporation
Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION W3
(Not for Publication)

TENTATIVE SPECIFICATION
February 6, 1964

TUNGSTEN ALLOY SHEET

1. GENERAL

1.1 This specification covers commercial or semi-commercial tungsten alloy sheet intended for structural fabrication by the tungsten arc and electron beam welding processes, and designated as follows:

P D Spec Designation	Description
W3-1	Tungsten -25% w/o Rhenium
W3-	

1.2 No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

2. MANUFACTURE

2.1 PROCESS: Material furnished to this specification shall be converted from electron beam and/or consumable electrode arc melted ingots.

2.2 CONDITION: The manufacturer shall furnish the sheet in the stress relieved condition and shall certify as to that condition per the requirements of Section 7.

3. CHEMICAL PROPERTIES AND TESTS

3.1 CHEMICAL COMPOSITION: The materials shall conform to the following composition:

Element	W3-1 Weight Per Cent
Rhenium	24.0 - 26.0
Carbon	0.005 max.
Oxygen	0.008 max.
Nitrogen	0.006 max.
Hydrogen	0.0005 max.
Tungsten	Remainder

Printed in U.S.A.

3.2 CHECK ANALYSIS: An analysis of each ingot shall be made by the manufacturer to determine the percentages of the elements specified in Table I. The chemical composition thus determined shall be reported to the purchaser and shall conform to the requirements specified in Section 3.1.

4. MECHANICAL PROPERTIES AND TESTS: Mechanical properties and tests will be specified on the purchase order.

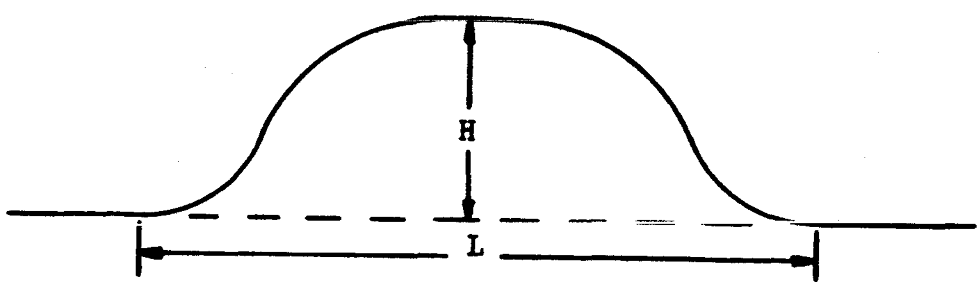
5. DIMENSIONS AND FINISH

5.1 WORKMANSHIP: Material shall be uniform in quality and condition, clean, sound, smooth, and free from "oil cans" of depth in excess of the flatness tolerances, ripples, foreign materials, and internal and external imperfections detrimental to fabrication or performance of the material.

5.2 SIZE: Where required, minimum sizes will be specified on the purchase order.

5.3 FLATNESS: Flatness tolerance shall not exceed five per cent as determined by the following formula:

$$\% \text{ Flatness} = \frac{H}{L} \times 100$$



Where: H is equal to the maximum distance between a flat reference surface and the lower surface of the sheet or plate, and L is equal to the minimum distance between points of contact of the sheet or plate with the flat reference surface.

If a general bow in the material can be eliminated by slight pressure without ends coiling or an "oil can" effect resulting, the material will be accepted.

6. OTHER REQUIREMENTS: When indicated on purchase orders, requirements not listed above or requirements more severe than those listed above which are referenced by a supplier's manufacturing specification will be adhered to.

7. TEST REPORTS: The manufacturer shall furnish five copies of a certified test report showing the results of the tests specified in Sections 3, 4, and 5 and bearing a statement as to the condition of the material as specified in Section 2.2. The report shall show the Purchase Order Number; P D Specification Number including dash number and sub letter; Per Cent of the Final Reduction; Temperatures of the Anneals Before and After the Final Reduction; Dimensions; Heat Number; and Name of Manufacturer.

8. PACKING AND MARKING

8.1 PACKING: The sheet or plate shall be packed in such a manner as to prevent injury during shipment.

8.2 MARKING: Each sheet or plate shall be marked showing the P D Specification and Dash Number; Heat Number; Manufacturer's Identification; Nominal Thickness in Inches. The marking shall be sufficiently stable to withstand ordinary handling.